Claims

25

1. A process for the preparation of an aromatic azo-compound 4

$$R^{1}_{m}$$
 $N=N$
 R^{2}_{n}

or a salt thereof,

comprising the step of treating aromatic amino-compounds 5 and 6

$$R_{m}^{1}$$
 NH_{2} R_{n}^{2} NH_{2}

with (i) hydrogen peroxide and acetic acid, followed by (ii) conc. sulphuric acid, to yield aromatic azo-compound 4 or a salt thereof, wherein

each m and each n is independently 0, 1, 2, 3, 4 or 5, and

each R¹ and each R² is independently an optionally substituted alkyl, alkenyl, alkynyl, aryl, arylalkyl, arylalkenyl, arylalkynyl, alkylaryl, alkenylaryl or alkynylaryl group which may include one or more heteroatoms N, O or S in its carbon skeleton, -F, -Cl, -Br, -I, -CF₃, -CCl₃, -CBr₃, -CI₃, -OH, -SH, -NH₂, -CN, -NO₂, -COOH, -R³-O-R⁴, -R³-S-R⁴, -R³-SO-R⁴, -R³-SO₂-R⁴, -R³-SO₂-OR⁴, -R³O-SO₂-R⁴, -R³-SO₂-N(R⁴)₂, -R³-NR⁴-SO₂-R⁴, -R³O-SO₂-OR⁴, -R³O-SO₂-N(R⁴)₂, -R³-NR⁴-SO₂-OR⁴, -R³-N(R⁴)₂, -R³-N(R⁴)₃, -R³-P(R⁴)₂, -R³-Si(R⁴)₃, -R³-CO-R⁴, -R³-CO-OR⁴, -R³O-CO-R⁴, -R³-CO-N(R⁴)₂, -R³-NR⁴-CO-R⁴, -R³O-CO-OR⁴, -R³O-CO-N(R⁴)₂, -R³-NR⁴-CO-N(R⁴)₂, -R³-CS-R⁴, -R³-CS-OR⁴, -R³O-CS-R⁴, -R³-CS-N(R⁴)₂, -R³-NR⁴-CS-R⁴, -R³O-CS-OR⁴, -R³O-CS-N(R⁴)₂, -R³-NR⁴-CS-N(R⁴)₂, all optionally protected, wherein

each $-R^3$ - is independently a chemical bond, a C_1 - C_{10} alkylene, C_1 - C_{10} alkenylene or C_1 - C_{10} alkynylene group, and

each $-R^4$ is independently hydrogen, unsubstituted C_1 - C_6 alkyl or unsubstituted C_6 - C_{10} aryl.

- 2. A process as claimed in claim 1, wherein the aromatic amino-compounds 5 and 6 are the same and together form a symmetric aromatic azo-compound 4.
- 3. A process as claimed in claim 1, wherein the aromatic amino-compounds 5 and 6 are different and together form an asymmetric aromatic azo-compound 4.
 - 4. A process for the preparation of 3,3'-azo-bis(6-hydroxybenzoic acid) 1

$$HO_2C$$
 $N=N$
 OH

or a salt or derivative thereof, comprising the step of treating a 5-amino salicyclic acid derivative 2a

or a salt or derivative thereof, with (i) hydrogen peroxide and acetic acid, followed by (ii) conc. sulphuric acid, to yield a 3,3'-azo-bis(6-hydroxybenzoic acid derivative) 1a

or a salt or derivative thereof, wherein

5

15

20

X is OR, SR or N(R)2,

when X is OR or SR, R is independently an optionally substituted alkyl, alkenyl, alkynyl, aryl, arylalkyl, arylalkenyl, arylalkynyl, alkylaryl, alkenylaryl or alkynylaryl group which may include one or more heteroatoms N, O or S in its carbon skeleton, hydrogen, -Si(alkyl), or -Sn(alkyl), and

when X is N(R)₂, each R is independently an optionally substituted alkyl, alkenyl, alkynyl, arylalkyl, arylalkynyl, arylalkynyl, alkylaryl, alkenylaryl or alkynylaryl group which may include one or more heteroatoms N, O or S in its carbon skeleton, hydrogen, -SO₂-(aryl), -NH₂, -NH(alkyl) or -NH(aryl), or both R together form an optionally substituted cycloheteroalkyl, cycloheteroalkenyl or heteroaryl group.

- 5. A process as claimed in claim 4, wherein X is OR and R is an optionally substituted alkyl, aryl or arylalkyl group.
- 6. A process as claimed in claim 5, wherein X is OR and R is an unsubstituted alkyl group.
- 7. A process as claimed in claim 6, wherein X is OR and R is an unsubstituted 15 C₁-C₆ alkyl group.
 - 8. A process as claimed in claim 7, wherein X is OR and R is methyl.

10

- 9. A process as claimed in claim 5, wherein X is OR and R is an optionally substituted arylalkyl group.
 - 10. A process as claimed in claim 9, wherein X is OR and R is benzyl.
- 11. A process as claimed in claim 4, further comprising a step of deprotecting the 3,3'-azo-bis(6-hydroxybenzoic acid derivative) 1a to yield 3,3'-azo-bis(6-hydroxybenzoic acid) 1 or a salt or other derivative thereof.
 - 12. A process as claimed in claim 11, wherein X is OR, and a 3,3'-azo-bis(6-hydroxybenzoic acid ester) 1b

$$RO_2C$$
 $N=N$
OH

is deprotected with sodium hydroxide to yield the disodium salt 1d of 3,3'-azo-bis(6-hydroxybenzoic acid)

$$Na^{+} O_2C$$
 $N=N$
 OH
 OH

13. A process as claimed in claim 12, wherein X is OR, both R are methyl, and a dimethyl-3,3'-azo-bis(6-hydroxybenzoate) 1c

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$$MeO_2C$$
 $N=N$
 OH

is deprotected with sodium hydroxide to yield the disodium salt 1d of 3,3'-azo-bis(6-10 hydroxybenzoic acid)

$$Na^+ O_2C$$
 $N=N$
 O_2
 O_2
 O_3
 O_4
 O_4

- 14. 3,3'-Azo-bis(6-hydroxybenzoic acid) 1 or a salt or derivative thereof, obtained by a process as claimed in claim 4.
- 15. Disodium salt 1d of 3,3'-azo-bis(6-hydroxybenzoic acid), obtained by a process as claimed in claim 4.
- 16. A pharmaceutical composition, comprising 3,3'-azo-bis(6-hydroxybenzoic 20 acid) 1 or a salt or derivative thereof as claimed in claim 14 and a pharmaceutically acceptable carrier or diluent.

- 17. A method of treating an inflammatory disease, comprising administering a pharmaceutically effective amount of 3,3'-azo-bis(6-hydroxybenzoic acid) 1 or a salt or derivative thereof as claimed in claim 14, to a subject in need of such treatment.
- 18. A method as claimed in claim 17, wherein the inflammatory disease is ulcerative colitis.